Laboratory Quality Assurance/Quality Control (Soil/Sediment) Samples

H.1.2 Polyaromatic Hydrocarbons

These samples were subcontracted by Torrent Laboratories to Calscience Environmental Laboratory in Garden Grove, CA. The samples underwent gel permeation chromatography (GPC) in order to remove an oily matrix and allow for lower detection limits

This section summarizes the results of the QA/QC review of the EPA Method 8270C SIM for Polyaromatic hydrocarbons (PAHs).

<u>Holding Time Review</u>. All extracted within 14 days of collection and were analyzed within 40 days of extraction except sample 0602119-013A (the subcontract laboratory did not always supply adequate sample cross reference tables, and, in some cases, Torrent log-in numbers are used in this section). The above referenced sample was analyzed after twice the expiration of the extraction holding time. Based on professional judgment, in the sample in question, all non-detects were rejected as not-usable and flagged "R" and all reported concentrations were flagged "J"

<u>Blank Review</u>. No equipment rinsate or field blanks were submitted. Method blanks did not contain any PAH compounds above the reporting limit.

<u>Surrogate Recovery</u>. The following table shows when surrogates were reported outside of their QC acceptance limits:

Sample	Surrogate	Percent Recovery	QC Acceptance Range		
02-B146-12.0	All	Above	N/A		
02-B30-03.0	p-Terphenyl-d14	184	23% to 160%		
02- D 30-03.0	2-Fluorobiphenyl	147	33% to 144%		
02-B53-04.0	2-Fluorobiphenyl	197	33% to 144%		
03-B05-00.5	p-Terphenyl-d14	180	23% to 160%		
	Nitrobenzene-d5	273	28% to 139%		
02-B32-00.5	p-Terphenyl-d14	941	23% to 160%		
	2-Fluorobiphenyl	261	33% to 144%		

When only one surrogate is out no action was taken. In the case of the three samples where two or more surrogates were recovered above the upper limit of their QC range, reported concentrations were flagged "J," estimated.

Matrix Spikes and Duplicates (MS/MSD). The following samples were spiked as MS/MSD samples: 02-B36-15.5, 06-008-B01-00.5, 0604107-014A, 02-B19-06.0, 02-B04-00.5, 0602119-013A. The following anomalies were observed:

Sample 06-008-B01-00.5 – The RPD between the MS and MSD recoveries of pyrene was 45%, which exceeded the QC limit of 20%. In that sample reported concentrations of benzo(a)pyrene and pyrene were flagged "J," estimated. This result also indicates a generally poor precision in this matrix.

Sample 0604107-14A – The RPD between the MS and MSD recoveries of pyrene was 44%, which exceeded the QC limit of 20%. Pyrene was not detected in the sample and the reporting limit was flagged "UJ," estimated. However this result suggests possible poor precision for pyrene and possibly benzo(a)pyrene and ideno(1,2,3-c,d) pyrene in all of the samples associated with this matrix.

Sample 02-B19-06.0 – MS/MSD recoveries were within QC limits for acenaphthene and pyrene, but the RPDs between the percent recoveries exceeded their QC limits (39% vs. QC limit of 11% for acenaphthene and 31% vs. QC limit of 20% for pyrene). Since these compounds were not present in the samples, no action was taken. These results do suggest the possibility of poor precision for these two compounds.

Sample 02-B04-00.5 – MS/MSD recoveries were within QC limits for pyrene, but the RPD between the percent recoveries of 25% exceeded the QC limit of 20%. The reported concentration of pyrene in the spiked sample was flagged "J," estimated. There is a possibility that other analytes in this matrix may be subject to poor precision.

Sample 0602119-013A – MS/MSD recoveries were within QC limits for pyrene, but the RPD between the percent recoveries of 32% exceeded the QC limit of 20%. Since the pyrene result had previously been qualified, no further action was required.

<u>Laboratory Control Samples (LCS)</u>. All LCS percent recoveries were within the QC acceptance range.

<u>Field Duplicate Sample Review</u>. There were no field duplicates submitted.

<u>Reporting Limits and Dilutions</u>. Samples were diluted in order to quantitate target compounds when the undiluted concentration exceeded the calibration curve. Generally the whole sample was diluted and non-detects was reported below reporting limits that were raised in proportion to the dilution

factors. In some cases the original non-detects were reported and only the high concentration compounds were reported from a dilution. The details are available in the individual validation reports.

H.1.2 Organochlorine Pesticides

This section summarizes the results of the QA/QC review of the EPA Method 8081A for organochlorine pesticides

<u>Holding Time Review</u>. Holding times of 7 days to extraction and 40 days to analysis were met for all samples

Method Blanks. No target analytes were reported in the method blanks.

<u>Surrogate Recovery</u>. Due to required dilutions, surrogates were diluted out of some samples. Results are not qualified when surrogates are diluted from samples. In the following instances the surrogate, dichloro biphenyl (DCB), was recovered outside of its 54.6% to 127% QC acceptance range:

Sample	DCB % Recovery
02-B29-0.3.5	163
06-009-B02-06.0	145
06-006-B01-06.0	178
06-005-B01-10.1	146
01-B16-00.5	51.0
01-B04-04.5	51.6
01-B14-00.5	138

When the percent recovery exceeded the QC acceptance range, reported concentrations were flagged "J," estimated. When the percent recovery was below the QC acceptance range, the reporting limits of the non-detects were flagged "UJ," estimated and the reported concentrations were flagged "J," estimated.

Matrix Spike and Duplicate (MS/MSD). The following samples were spiked as MS/MSDs: 05-B03-03.5, 02-B04-00.5, 05-B03-03.5, 02-B19-06.0, 01-B01-04.0, 02-B04-00.5, and 02-B36-15.5. The following anomaly was observed:

Sample 02-B04-00.5 – Endrin and aldrin were not recovered in the MS (0%) and their mean percent recoveries of 22.8% and 24.8%, respectively, were below their QC acceptance ranges. In the spiked sample, neither analyte was reported and the reporting limits associated with the

non-detects were flagged "UJ," estimated. These low recoveries may indicate a negative bias for reported concentrations of these analytes in this matrix.

<u>Laboratory Control Samples (LCS)</u>. All LCS compounds were recovered within their QC acceptance range.

Field Duplicate Sample Review. There were no field duplicates submitted.

<u>Reporting Limits and Dilutions</u>. Due to the presence of hydrocarbons in the samples, dilutions by factors between 2 and 125 were required. Reporting limits were increased in proportion to these dilutions.

H.1.3 Polychlorinated Biphenyls (PCBs)

This section summarizes the results of the QA/QC review of the EPA Method 8082 for PCBs.

<u>Holding Time Review</u>. Holding times of 14 days to extraction and 40 days to analysis were met for all samples

Method Blanks. PCBs were not reported in the method blanks above their reporting limits.

<u>Surrogate Recovery</u>. In the following instances the surrogate, DCB, was recovered outside of its 63.7% to 126% QC acceptance range:

Sample	DCB % Recovery
02-B41-00.5	57.2
02-B52-00.5	57.5
02-B33-17.5	59.2
02-B31-20.0	62.7
03-B04-11.0	62.5
03-B07-00.5	60.6
03-B08-15.0	62.5
03-B09-07.0	54.6
03-B01-07.5	50.9
03-B06-07.5	61.9

Reporting limits of non-detects in these samples were flagged "UJ," estimated and reported concentrations were flagged "J," estimated.

Matrix Spike and Duplicate (MS/MSD). Samples 02-B36-15.5, 05-B06-00.5, and 02-B04-015. None of the percent recoveries or RPDs between the percent recoveries were outside of their QC acceptance range.

<u>Laboratory Control Samples (LCS)</u>. Both PCBs were recovered within their QC acceptance range.

<u>Field Duplicate Sample Review</u>. There were no field duplicates submitted.

<u>Reporting Limits and Dilutions</u>. Due to the oily matrix a number of samples required dilutions by factors of five. Reporting limits were increased by the same factor.

H.1.4 Lead

This section summarizes the results of the QA/QC review of the EPA Method 6010B for lead.

<u>Holding Time Review</u>. Holding time limit of 180 days for lead was met.

Method Blanks. Lead was not found in any of the method blanks.

Matrix Spike and Duplicate (MS/MSD). 02-501 (Lead and Arsenic), 03-B15-15.0, 07-B01-00.5, 06-010-B01-00.5, 06-006-B01-00.5, 03-B07-00.5, 02-B28-00.5, 02-B41-16.0, 04-B04-03.5, 03-B15-00.5, 05-B01-00.5, 02-B58-00.5, 03-B13-11.5, 02-B12-00.5, 02-B51-04.5, 02-B16-02.0, and 02-B03-06.0. The following anomalies were noted (the QC acceptance range is 75% to 125% and the RPD limit is 30%.

Spiked Sample	Mean Lead Percent Recovery	RPD (if above 30%)
03-B15-00.5	192	45.8
07-B01-00.5	62.6	N/A
06-010-B01-005	312	76.2
06-006-B01-00.5	32.8	N/A
03-B07-00.5	27.9	N/A
02-B28-00.5	146.2	N/A
02-B58-00.5	125.1	N/A
03-B13-11.5	943	>>30%

In all samples associated with the MS/MSDs, when the mean percent recovery was below 75%, the reporting limit was flagged "UJ," estimated and reported concentrations were flagged "J," estimated. When the percent recovery exceeded 125%, reported concentrations of lead in the associated samples were flagged "J," estimated. See the original validation reports to see the samples affected by each MS/MSD.

<u>Laboratory Control Samples (LCS)</u>. Lead was recovered within the QC acceptance range of 80% to 120%.

<u>Field Duplicate Sample Review</u>. There were no field duplicates submitted.

<u>Reporting Limits and Dilutions</u>. Samples 03-B12-07.0 and 03-B14-07.0 were diluted by factors of four in order to quantitiate lead. The reporting limits were increased by the same factor. The reported concentrations of lead in these samples exceeded the elevated reporting limits.

H.1.5 CAM 17 Metals

This section summarizes the results of the QA/QC review of the EPA Method 6010B for the CAM 17 Suite of Metals (EPA Method 7471 for Mercury).

<u>Holding Time Review</u>. Holding time limits of 28 days for mercury and 180 days for all other metals were met.

<u>Method Blanks</u>. There were no CAM 17 metals present in the method blanks above their reporting limits

<u>Matrix Spike and Duplicate (MS/MSD)</u>. The following samples were spiked for MS/MSD analyses: 06-033-B01-05.0 (Hg only), 03-B15-00.5, 07-B01-00.5, 05-B01-00.5, 02-B41-16.0, 04-B0-3.5, 01-B03-00.5, 01-B10-00.5, 05-B01-00.5, 02-B12-00.5, 02-B51-04.5, 02-B16-02.0, 02-B17-09.0 (Hg only), 02-B08-00.5 (Hg only), 06-008-B01-00.5, 06-006-B01-06.0, 06-010-B01-10.0, 03-B07-00.5, 03-B04-00.5, 02-B24-005 (Hg not included), 02-B28-00.5. The following anomalies were noted (the QC acceptance range is 75% to 125% and the RPD limit is 30%.

Spiked Sample	Metal	Percent Recovery	RPD (if above 30%)		
	Antimony	32.0	N/A		
03-B15-00.5	Copper	147.5	N/A		
	Lead	192	45.8		
07-B01-00.5	Antimony	28.2	N/A		
07-001-00.3	Lead	62.8	N/A		
	Antimony	30.2	N/A		
05-B01-00.5	Nickel	73.0	N/A		
	Thallium	71.0	N/A		

Spiked Sample	Metal	Percent Recovery	RPD (if above 30%)			
02-B41-16	Antimony	60.6	N/A			
U2- D 41-10	Barium	125.5	N/A			
04-B04-3.5	Arsenic	64.3	N/A			
U4-DU4-3.3	Nickel	138	N/A			
01-B03-00.5	Lead	229	95			
01-603-00.3	Zinc	119	31			
02-B12-00.5	Nickel	70.4	N/A			
02-B51-04.5	Antimony	72.1	N/A			
02-031-04.3	Nickel	137.7	N/A			
02-B16-02.0	Antimony	72.3	N/A			
02-610-02.0	Nickel	63.6	N/A			
	Antimony	56.0	N/A			
06-006-B01-06.0	Lead	32.8	N/A			
	Barium	69.3	N/A			
	Antimony	30.1	N/A			
06-010-B01-10.0	Lead	325	N/A			
00-010-д01-10.0	Barium	135	N/A			
	Zinc	152	N/A			
	Lead	27.9	N/A			
03-B07-00.5	Zinc	70.0	N/A			
	Nickel	71.5	N/A			
	Zinc	70.9	N/A			
	Nickel	67.3	N/A			
03-B04-00.5	Antimony	72.8	N/A			
	Chromium	71.4	N/A			
	Barium	N/A	72.4%			
	Barium	31.5	N/A			
02-B28-00.5	Lead	127.2	N/A			
	Zinc	146.2	N/A			

When recoveries were below 75%, reporting limits of non-detects were flagged "UJ," estimated and reported concentrations were flagged "J," estimated. All samples associated with the MS/MSD were affected. Then the recovery exceeded 125%, reported concentrations were flagged "J," estimated. Samples associated with the MS/MSDs are listed in the separate validation reports.

Laboratory Control Samples (LCS). The following samples were associated with an LCS recovery of mercury of 122%, which exceeded the QC acceptance range of 80% to 120%: 06-007-B01-04.0, 06-007-B03-07, 06-008-B01-00.5, 06-008-B03-07.0, 06-006-B01-06.0, 06-010-B01-10.0, 06-010-B02-07.0, 06-010-B03-03.0, and 06-010-B04-09.0. In each of these samples, positive detections of mercury were flagged "J," estimated.

<u>Field Duplicate Sample Review</u>. There were no field duplicates submitted.

<u>Reporting Limits and Dilutions</u>. Individual metals were run at higher dilutions in order to quantititate specific metals. In each case the reporting limits were increased in proportion to the dilution factors. Reported concentrations of these metals exceeded the elevated reporting limits.

H.1.6 Total Recoverable Petroleum Hydrocarbons

This section summarizes the results of the QA/QC review of the Standard Method SM 5520M for total recoverable petroleum hydrocarbons (TRPH).

<u>Holding Time Review</u>. Samples were all analyzed within the required holding time.

Method Blanks. TRPH was not present in any of the method blanks.

Matrix Spike and Duplicate (MS/MSD) There were no samples spiked for MS/MDS analysis.

Laboratory Control Samples (LCS). All LCS recoveries were within QC acceptance limits.

Field Duplicate Sample Review. There were no field duplicates submitted.

Reporting Limits and Dilutions. No dilutions were required.

H.1.7 Dioxins and Furans

These samples were subcontracted by Torrent Laboratories to Columbia Analytical Services in Houston, TX.

This section summarizes the results of the QA/QC review of the EPA Method 8290 for Dioxins and Furans.

Samples, in this report, are referred to using their Columbia Analytical Services (CAS) login numbers.

<u>Holding Time Review and Arrival Condition:</u> All samples arrived at the laboratory at temperatures between 11° C and 23° C, which were significantly above the 6° C upper limit. Since dioxins and furans are extremely stable compounds, no actions were taken and the data were not qualified.

<u>Blank Review:</u> In all sample delivery groups a number of target compounds were reported in the method blank at low concentrations, such that the five times the blank concentration was less than the sample concentration of the same compound. Consequently the compound was reported present in the sample and was flagged "B" by the laboratory. On the case of OCDF in sample E0600334-001 and -002 and five analytes in sample E0600147-002 (1,2,3,4,6,7,8-HpCDD; OCDD; 1,2,3,4,6,7,8-HpCDF; OCDF, and Total Hepta-Dioxins, five times the blank concentration exceeded the sample concentration. The result was flagged "U" and should be treated as a non-detect below the originally reported concentration.

<u>Labeled Compounds and Cleanup Standard Recoveries</u>: In the following samples, the listed labeled compounds were recovered outside their QC acceptance ranges of 40% to 135%:

Sample	Compound	% Recovery
	13C-1,2,3,4,6,7,8-HpCDD	39.3
E0600126-001	13C-OCDD	22.2
	13C-1,2,3,4,6,7,8-HpCDF	37.9
	13C-1,2,3,4,6,7,8-HpCDD	36.5
E0600126-003	13C-OCDD	20.4
	13C-1,2,3,4,6,7,8-HpCDF	37.4
E0600126-005	13C-OCDD	38.9
E0600126-006	13C-OCDD	37.0
E0600126-009	13C-OCDD	36.4
E0602127-016	13C-OCDD	34.2
E0600126-017	13C-2,3,7,8-TCDD	38.2
	13C-1,2,3,7,8-PeCDD	39.3
	13C-2,3,7,8-TCDF	39.3
	13C-1,2,3,7,8-PeCDF	37.6
	13C-1,2,3,4,7,8-HxCDF	177.8
E0600336-001	13C-OCDD	22.8
	13C-1,2,3,4,6,7,8-HpCDF	36.7

Sample	Compound	% Recovery
E0600334-001	13C-OCDD	28.7
E0600334-002	13C-OCDD	35.8

Reported concentrations of these compounds, in the associated samples, were flagged "J", estimated.

<u>Matrix Spikes and Duplicates (MS/MSD)</u>: Only one sample was spiked for MS/MSD analysis, sample E0600141-020. The following compounds had mean percent recoveries above the 70% to 130% QC limit:

H.1.2 Compound	% Recovery
2,3,7,8-TCDF	280
1,2,3,4,7,8-HxCDF	160
1,2,3,6,7,8-HxCDF	140
2,3,4,6,7,8-HxCDF	133
1,2,3,7,8,9-HxCDF	139
1,2,3,4,6,7,8-HpCDF	218
1,2,3,4,7,8,9-HpCDF	140

Reported concentrations of these compounds in sample E0600141-020 were flagged "J". However the results should be interpreted that, at least with this sample, the matrix effect is for a positive bias.

<u>Laboratory Control Samples (LCS)</u>: All LCS percent recoveries were within the QC acceptance range of 70% to 130%.

<u>Initial Calibrations</u>: Percent valley for 2,3,7,8-TCPP, percent standard deviation, signal to noise ratios and ion abundance ratios were checked. All initial calibration criteria were met. In the case of OCDD in sample E0600147-006, the reported concentration of 2,600 ng/kg exceeded the instrument calibration range and was estimated and flagged "J".

Continuing Calibrations: All of the samples undergoing Level 3 validation met the following QC criteria: % valley for 2,3,7,8-TCPP, percent difference (%D) between the IC and CCV response, signal to noise and ion abundance ratios. In the case of the Level 4 validation there were a number of anomalies noted. The %Ds for the closing standard on March 15, 2006 at 15:02 for 1,2,3,6,7,8-HxCDF and 1,2,3,7,8,9-HxCDF were 22% and 23% respectively. Per Method 8290 Section 8.3.2.4, PCDDs/PCDFs results for samples analyzed on March 15, 2006

were quantified using the average response factors from the initial and closing calibration verifications for that day rather than the average response factors from the initial calibration. This alternate method of quantitation accounts for the observed instrument drift and data qualification was not considered necessary.

The %D for the opening standard on March 24, 2006 at 10:36 for OCDF was 23%. The %Ds for the closing standard on March 24, 2006 at 20:40 for 1,2,3,6,7,8-HxCDF and OCDF were 22% and 23% respectively. Per Method 8290 Section 8.3.2.4, PCDDs/PCDFs results for samples analyzed on March 24, 2006 were quantified using the average response factors from the initial and closing calibration verifications for that day rather than the average response factors from the initial calibration. This alternate method of quantitation accounts for the observed instrument drift and data qualification was not considered necessary.

Ion abundance ratios met method criteria for the calibration verification standard analyses and the S/N for all compound peaks was >10:1. Calibration verification criteria were met and data qualification was not necessary.

<u>Field Duplicate Sample Review</u>: There were no field duplicates submitted.

Reporting Limits and Dilutions: Samples were not diluted either due to the matrix or as required to quantitiate target compounds. All concentrations that were reported below the reporting limit (minimum level) were flagged "J", estimated by the laboratory and these flags were carried over in the validation.

The following additional items were part of an extended Level 4 validation and only apply to the following samples: E0600147-002, E0600147-003, E0600147-004, E0600147-005 and E0600147-006.

Instrument Stability: Instrument stability criteria were met and data were not qualified.

Mass Calibration and Mass Spectrometer Resolution: Both PFK criteria were met. The resolution exceeded 10,000 at m/z 304.9824 and the deviation between the exact and the oretical mass must be less than 5 ppm.

Window Defining Mixture: Both the frequency and retention time requirements were met for the performance check solution.

Chromatographic Resolution: The 2,3,7,8-TCDD and its close eluters meeting the specified maximum 25% valley criterion demonstrated the minimum separation on the DB-5 column.

Identification Criteria: For a gas chromatographic peak to be identified as a PCDD or PCDF, it must meet the following criteria:

- Retention times for 2,3,7,8-substituted congeners with an isotopically labeled internal standard must be within -1 to +3 seconds of the isotopically labeled internal standard,
- Retention times for 2,3,7,8-substitued congeners without an isotopically labeled standard must be within 0.005 retention time units of the relative retention times measured in the routine calibration (calibration verification),
- The ion current responses for both ions used for quantitation purposes for both unlabeled and labeled compounds must maximize simultaneously (± 2 seconds),
- The ion abundance ratios must meet the criteria contained in Table 8 of the method (\pm 15% of theoretical).
- The ion current intensities (signal) for target compounds must be ≥ 2.5 times the noise level (S/N ≥ 2.5),
- The polychlorinated diphenyl ether (PCDE) channel must have no PCDE present (defined as a $S/N \ge 2.5$ within 2 seconds of the retention time of the target analyte).

No errors in compound identification were found and data qualification was not necessary. Results reported as Estimated Maximum Possible Concentrations (EMPCs) and the resulting data qualification are discussed and summarized in Section 3.15 below.

Toxicity Equivalent Factor (TEF) and Isomer Specificity: Concentrations for the 2,3,7,8-substituted tetra- trough octa- isomers were multiplied by their respective Toxicity Equivalence Factors (TEFs) to determine the 2,3,7,8-TCDD toxicity equivalence concentration (TEQ). CAS-Houston used the World Health Organization (WHO) adopted TEFs taken from: Van Den Berg, et al: Toxicity Equivalency Factors (TEFs) for PCBs, PCDDs, PCDFs, for Humans and Wildlife, 1998. The WHO TEFs differ from the TEFs listed in SW-846 Method 8290 Table 10 for the following congeners:

Congener	WHO 1998 TEF	Method 8290 TEF
1,2,3,7,8-PeCDD	1	0.50
OCDD	0.0001	0.001
OCDF	0.0001	0.001

TEFs listed in Method 8290 Table 10 are taken from *Interim Procedures for Estimating Risks* Associated with Exposures to Mixtures of Chlorinated Dibenzo-p-Dioxin and Dibenzo-furans (CDDs and CDFs) and 1989 Update.

TEQ concentrations were calculated using the TCDF values reported from the quantitation analyses performed using the DB-225 column. Chromatographic separation on the DB-225

column was demonstrated for 2,3,7,8-TCDF and its close eluters by meeting the method-specified maximum 25% valley criterion.

Also, CAS-Houston included EMPCs in the TEF calculations. Functional Guidelines Section XII states that EMPCs are not to be included in the TEF calculations. Therefore, the TEQ concentrations reported by CAS-Houston are higher for samples including EMPC values than if TEQ concentrations were calculated excluding EMPCs as Functional Guidelines states.

<u>Second Column Confirmation</u>: Second column quantitation analyses were performed using the DB-225 column for 2,3,7,8-TCDF if this congener was detected on the DB-5 column. Chromatographic separation on the DB-225 column was demonstrated for 2,3,7,8-TCDF and its close eluters by meeting the method-specified maximum 25% valley criterion. For this reason, sample 2,3,7,8-TCDF results subjected to Level IV validation were selected for use from the DB-225 column analyses.

<u>Estimated Detection limit and Estimated Possible Concentration:</u> Sample specific Estimated Detection Limits (EDLs) are estimates made by the laboratory of the concentration of a given analyte that must be present to produce a signal with a peak height of at least 2.5 times the background noise signal level. The estimate is specific to a particular analysis of the sample and is affected by sample size, dilution, etc. Congener specific EDLs were calculated and reported for each sample.

Results not meeting qualitative identification criteria specified in the bulleted list below were reported as EMPCs with concentrations calculated as specified by the method. Examination of the preparation log showed that method-required cleanup steps (sulfuric acid and silica and carbon column cleanups) were performed. Thus, interferences resulting in failure to meet the identification criteria are not considered attributable to failure to perform method-required cleanup procedures.

The reviewer checked the SICPs for Polychlorinated Diphenyl Ether (PCDPE) interferences to the quantitation of PCDFs for those PCDFs that were reported as detected. Any detected sample result potentially influenced by PCDPE interference would have been reported by the laboratory as an EMPC. No sample results were reported as EMPCs due to PCDPE interference.

Results for analytes meeting the following identification criteria specified in the method:

- Retention times (Section 7.8.4.1),
- Signal to noise ratio (Section 7.8.4.3),
- Lack of PCDPE interferences (Section 7.8.4.4),

But not the following criteria:

• Ion abundance ratio (Section 7.8.4.2),

Were qualified as estimated (J-E) at the EMPC due to failure to meet these qualitative identification criteria. The associated bias direction for the EMPC is considered to be high. For these results, the reported concentration is considered to be the "effective" detection limit. The table below lists the affected sample results.

Results Qualified as EMPCs due to Identification Criteria

Lab ID Number	2,3,7,8-TCDD	1,2,3,7,8-PeCDD	1,2,3,4,7,8-HxCDD	1,2,3,6,7,8-HxCDD	1,2,3,7,8,9-HxCDD	1,2,3,4,6,7,8-HpCDD	осрр	2,3,7,8-TCDF	1,2,3,7,8-PeCDF	2,3,4,7,8-PeCDF	1,2,3,4,7,8-HxCDF	1,2,3,6,7,8-HxCDF	2,3,4,6,7,8-HxCDF	1,2,3,7,8,9-HxCDF	1,2,3,4,6,7,8-HpCDF	1,2,3,4,7,8,9-HpCDF	OCDF
E0600147-002										X	X						
E0600147-003		X		X	X											X	
E0600147-004	X	X									·				•		
E0600147-005		X	X		X						·				•		•
E0600147-006															•		•

Sample Quantitation and Results Verification: Target compound quantitation was evaluated by recalculating approximately 10% of the reported results to verify that calculations were performed using the proper values for all factors in the calculation. These factors include target analyte selected ion current profile (SICP) peak areas, internal standard areas, internal standard concentrations, sample weights, sample moisture content, and initial and continuing calibration relative response factors (RRFs). No errors in sample quantitation were found and data qualification was not necessary. Additionally, the reviewer verified the calculation of a 2,3,7,8-TCDD toxicity equivalence concentration (TEQ) from TEFs and sample concentrations, and the calculation of EDLs and EMPCs. No errors in these calculations were found and data qualification was not necessary.

The reviewer checked for correspondence between the raw sample data and the summary data provided. No transcription or reporting errors were found and data qualification was not necessary.

All detectable results were reported within the ICAL range with one exception. The OCDD result for sample E0600147-006 of 2,536 nanograms per kilogram (ng/kg) exceeded the instrument calibration range and was therefore qualified as estimated (J).

Sample results, and EDLs were reported in the correct units. Sample results reported as detected below the reporting limit (minimum level) for PCDDs and PCDFs were qualified as estimated.

H.1.8 CAM 17 Metals – TCLP Extract

This section summarizes the results of the QA/QC review of the EPA Method 6010B for the CAM 17 Suite of Metals (EPA Method 7470A for Mercury).

<u>Holding Time Review</u>. In all cases, mercury extraction took place after twice the 28-day holding time for mercury analysis. All non-detects of mercury were flagged "R," rejected, and are not usable. Reported concentrations of mercury were flagged "J" < estimated.

<u>Method Blanks</u>. The method blank associated with samples 03-B09, 02-B46, 03-B04, and 04-B02 contained barium and molybdenum at concentrations of 0.02 mg/L and 0.01 mg/L, respectively. The reported concentration of molybdenum in sample 04-B02 was flagged "U" and should be treated as a non-detect below a reporting limit equal to the originally reported concentration.

<u>Matrix Spike and Duplicate (MS/MSD)</u>. Samples 02-B01, and 03-B09 were spiked as MS/MSD samples. The following anomalies were noted:

Sample 02-B01: Mercury had a mean recovery at 54.7%, which is below the 75% to 125% QC acceptance range. Since mercury in the samples associated with this MS/MSD were previously rejected, no further action was required.

Sample 03-B09: Zinc had a mean recovery of 59.2%, which is below the QC acceptance range of 75% to 125%. Inn the associated samples, 03-B09, 03-B46, 03-B04 and 04-B02 reported concentrations of zinc wee flagged "J," estimated.

<u>Laboratory Control Samples (LCS)</u>. All metals were recovered within their QC acceptance range of 80% to 120%.

Field Duplicate Sample Review. There were no field duplicates submitted.

Reporting Limits and Dilutions. No dilutions were required.

Laboratory Quality Assurance/Quality Control

Water samples include groundwater and surface water

H.2.1 Polyaromatic Hydrocarbons

<u>Holding Time Review</u>. All extracted within 14 days of collection and were analyzed within 40 days of extraction.

<u>Blank Review</u>. No equipment rinsate or field blanks were submitted. Method blanks did not contain any PAH compounds above the reporting limit.

Surrogate Recovery. Surrogates were recovered within their QC acceptance limits

<u>Matrix Spikes and Duplicates (MS/MSD)</u>. Water samples were not spiked for MS/MSD analysis by the laboratory.

Laboratory Control Samples (LCS). All LCS percent recoveries were within the QC acceptance range.

Field Duplicate Sample Review. There were no field duplicates submitted.

<u>Reporting Limits and Dilutions</u>. Water samples were not diluted and reporting limits were not raised.

H.2.2 Organochlorine Pesticides

This section summarizes the results of the QA/QC review of the EPA Method 8081A for organochlorine pesticides

<u>Holding Time Review</u>. Holding times of 7 days to extraction and 40 days to analysis were met for all samples

<u>Method Blanks</u>. No target analytes were reported in the method blanks.

<u>Surrogate Recovery</u>. The surrogate, DCB, was recovered below the 52% to 116% QC acceptance range for the following samples: 02-B31-W (50.7%), 02-B33-W (40%), 02-B18-W (35.5%), and was also below the QC acceptance range in the case of samples 02-B01-W and 02-B09-W (the validation reports did not give the percent recoveries for these two samples). In

all of the above samples reported concentrations of target analytes were flagged "J," estimated, and the reporting limits associated with non-detects were flagged "UJ," estimated.

In the case of samples SW-I-04, SW-I-01, SW-O-02 and SW-O-01, the surrogate, DCB, was not recovered (0%). The non-detects for these samples were flagged "R," rejected, and are not usable.

<u>Matrix Spike and Duplicate (MS/MSD)</u>. None of the water samples analyzed for organochlorine pesticides were spiked for MS/MSD analysis.

<u>Laboratory Control Samples (LCS)</u>. All LCS compounds were recovered within their QC acceptance range.

Field Duplicate Sample Review. There were no field duplicates submitted.

<u>Reporting Limits and Dilutions</u>. No solutions were required and reporting limits did not need to be raised.

H.2.3 Polychlorinated Biphenyls (PCBs)

This section summarizes the results of the QA/QC review of the EPA Method 8082 for PCBs.

<u>Holding Time Review</u>. Holding times of 7 days to extraction and 40 days to analysis were met for all samples

Method Blanks. PCBs were not reported in the method blanks above their reporting limits.

<u>Surrogate Recovery</u>. The surrogate DCB was recovered at 131%, which exceeds its QC acceptance range of 63.7% to 126%. Since all results were non-detects and since the recovery was high, no data were qualified.

Matrix Spike and Duplicate (MS/MSD). Samples WP-WELL, OS-O-02, 02-B12-W, and 03-B31-W were spiked as MS/MSD samples. None of the PCBs were outside of their QC acceptance range

<u>Laboratory Control Samples (LCS)</u>. All both PCBs were recovered within their QC acceptance range.

<u>Field Duplicate Sample Review</u>. There were no field duplicates submitted.

Reporting Limits and Dilutions. No dilutions were required.

H.2.4 CAM 17 Metals

This section summarizes the results of the QA/QC review of the EPA Method 6010B for the CAM 17 Suite of Metals (EPA Method 7470 for Mercury).

<u>Holding Time Review</u>. Holding time limits of 28 days for mercury and 180 days for all other metals were met.

Method Blanks. The method blank associated with samples 02-B21-W and 02-B12-W contained a low concentration of antimony (0.011 mg/L). This concentration was too small to affect any field sample results.

Matrix Spike and Duplicate (MS/MSD). Samples SW-I-049 (not for Hg), 02-B21-W, 02-B02-W, 02-B31-W and WP-WELL were spiked for most of the CAM 17 metals. Samples 03-B09-W and SW-I-01 were spiked for Hg only. All percent recoveries were between the QC acceptance limits of 75% to 125% and RPDs between the percent recoveries were below the QC limit of 20%.

<u>Laboratory Control Samples (LCS)</u>. All metals were recovered within their QC acceptance range of 80% to 120%.

<u>Field Duplicate Sample Review</u>. There were no field duplicates submitted.

Reporting Limits and Dilutions. No dilutions were required.